USEPA REGION 9 LABORATORY
RICHMOND, CALIFORNIA

STANDARD OPERATING PROCEDURE 312
MAINTENANCE AND TRACKING OF CANISTERS AND FLOW CONTROLLERS

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1 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) applies to the maintenance, cleaning, and certification of specially prepared canisters and flow controllers used for collection of air samples for volatile organic compound analysis. Procedures for tracking status and location of the equipment are provided. Technical aspects of the SOP are based on requirements in EPA Compendium Method TO15 *The Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)*. Deviations from the reference method are listed in Appendix A.

Canister types include SUMMA™ polished interior, SilcoSteel® silica coated interior, TO-Can® electro-polished interior, and Silonite® coated canisters.

2 METHOD SUMMARY

The Entech Model 3100A Canister Cleaning System cleans, leak tests, and evacuates up to six 6-L, twenty 1-L canisters, or twenty 400-mL canisters at a time. The procedure uses heat and humidified nitrogen flow to remove contaminants from the canisters. Canisters are certified by pressurizing with zero grade air or nitrogen and analyzing for contaminants. An aliquot is analyzed according to EPA Region 9 Laboratory SOP 311 *Analysis of Volatile Organic Compounds in Air and Soil Vapor* or EPA Region 9 Laboratory SOP 314 *Low Level Analysis of Volatile Organic Compounds in Air by GC/MS Selected Ion Monitoring* on a project-specific basis (i.e. each canister is certified using the SOP applicable to the project that will use the clean canister) to verify the absence of target analytes.

The Entech system is also used to clean flow controller parts and restrictors. Flow controllers are calibrated by attaching a flow meter to the inlet while the outlet is connected to a canister under vacuum.

The status and location of canisters and flow controllers is tracked using an Access database. The database is maintained by shipping, sample receiving, and analytical staff to maintain an up-to-date status of all equipment.

3 DEFINITIONS

A list of terms and definitions specific to this procedure appears below. For terms and acronyms in general use at the EPA Region 9 Laboratory refer to Appendix A of the Laboratory Quality Assurance Plan.

Cleanup batch: group of cans which are loaded in the cleaning oven and cleaned in the same cleaning sequence. For 400-mL or 1-L cans the batch may contain up to 20 cans which is the maximum the cleaning oven can hold. For 6-L cans the batch may contain up to six cans which is the maximum the cleaning oven can hold.

psia: pounds per square inch absolute, the pressure relative to a vacuum. Atmospheric pressure at sea level is 14.7 psia which is equal to 0 psig.

psig: pounds per square inch gauge, the pressure relative to atmospheric pressure.

4 SAFETY & HEALTH

All laboratory personnel must follow health and safety requirements outlined in current versions of the EPA Region 9 Laboratory Chemical Hygiene Plan and the Region 9 Laboratory Business Plan. Potential hazards specific to this SOP as well as pollution prevention and waste management requirements are described in the following sections.

4.1 Chemical Hazards

Due to the unknown and potentially hazardous characteristics of samples, all sample handling and preparation should be performed in a well-vented properly operating laboratory fume hood.

The toxicity and carcinogenicity of each reagent used in this method may not be fully established. Each chemical should be regarded as a potential health hazard and exposure to them should be minimized by good laboratory practices. Refer to the Safety Data Sheets located in Room 118 (library) and the LAN at I:\MSDS IMAGES for additional information.

4.2 Equipment and Instruments

Follow the manufacturer's safety instructions whenever performing maintenance or troubleshooting work on equipment or instruments. Unplug the power supply before working on internal instrument components. Use of personal protective equipment may be warranted if physical or chemical hazards are present.

Specially-prepared canisters should never be pressurized beyond the maximum allowable pressure of 45 psia.

5 SAMPLE CANISTER AND FLOW CONTROLLER TYPES AND STATUS

5.1 Canister types and volumes required for each analysis (SIM or full scan) are provided in EPA Region 9 Laboratory SOP 311 and SOP 314 or on a project-specific basis.

This SOP applies to cleaning of the following general types of canisters (hereafter referred to as "canisters" without specifying type):

- SUMMA Canister: Spherical stainless steel container that has undergone a SUMMA treatment that results in a highly polished interior surface resistant to adsorption of volatile organic compounds.

- SilcoSteel Canister: Spherical stainless steel container with fused silica interior lining that resists adsorption of volatile organic compounds. Purchased from Restek Corp or equivalent.

- Silonite Canister: Spherical stainless steel canister coated with Silonite that resists adsorption of volatile organic compounds. Purchased from Entech Instruments, Inc. or equivalent.

- TO-Can Canister: Spherical stainless steel container with an electro-polished and deactivated interior surface that resists adsorption of volatile organic compounds. Purchased from Restek Corp or equivalent.

5.2 Flow controllers: the two types of flow controllers used in the laboratory are described below:

- Critical Orifice Flow Controller: High purity flow regulation system used to fill canisters at a constant rate using a restrictor.

- Time Integrated Flow Controller: High purity flow regulation system used to fill canisters at a constant rate over a given period of time (i.e. 4, 8, 12, and 24 hours) using a vacuum controller body and a restrictor.

5.3 Tracking status and location

The status and location of all air canisters and flow controllers are tracked in the Canister Database (refer to Appendix D). Briefly, the process is as follows:

1. New canisters and flow controllers are entered into the database using unique serial numbers. Initial status is "To be cleaned."
2. Canisters and flow controllers are cleaned and certified and the status entered into the Canister Database.
3. A cleaned canister with its certificate of analysis is pulled for shipment. Flow controllers are selected at the same time. The analyst completes the pre-shipment checks.
4. The Sample Custodian updates the database upon shipment and again upon receipt of canisters and flow controllers. Analysis of the air sample is

completed according to the appropriate analytical SOP (including sample custody requirements).

5. After an air sample has been analyzed and storage of the sample for the time period outlined in EPA Region 9 Laboratory SOP 311 and/or SOP 314 is complete, the analyst updates the status of the canister in the Canister Database to "To Be Cleaned."

6 INTERFERENCES

Ambient air leaking into canisters, solvent contamination of any part of the system, and impurities in reagent gases are potential interferent.

6.1 Contamination from the ambient air in the laboratory is a concern. Canister cleaning and analytical systems are located in Room 203, which is maintained under positive pressure to prevent intrusion of volatile contaminants from surrounding areas.

6.2 Impurities in the dilution gas, organic compounds out-gassing from the cleaning system, and solvent vapors in the laboratory may result in contamination problems. To minimize this possibility, the cleaning apparatus should be assembled with clean, high quality components.

6.3 Nitrogen and air should be of high purity. Nitrogen and air should be humidified with organic free water that has been prepared according to EPA Region 9 Laboratory SOP 205 *Preparation of VOC-Free Method Blank Water*.

6.4 Canisters should be capped tightly with the valve closed during storage and shipment to minimize the chance of leakage and contamination.

6.5 Solvents and other compounds that are target analytes must never be introduced into the laboratory where volatiles analysis is performed. Dichloromethane, acetone, and other target compounds must be excluded from Rooms 201 and 203.

7 APPARATUS AND MATERIALS

This section describes recommended apparatus and materials to be used for the analysis. All equipment, reagents, and supplies must meet the technical and QC requirements of the reference method. Substitutions may be made provided that they are documented and equivalency is maintained.

7.1 Equipment

The canister cleaning system at the EPA Region 9 Laboratory is assembled from the following components:

- Entech Model 3100A Canister Cleaning System with built in molecular drag pump, humidification chamber, and pressure sensor. The system must be capable of evacuating a canister to an absolute pressure of <30 mTorr.

- L-C Oven (Lab-line Model 3513ENT) and stainless steel manifold with six connections for simultaneously cleaning multiple 6-L canisters.

- L-C Oven (Lab-line Model 3512) and stainless steel manifold with 20 connections for simultaneously cleaning multiple 1-L or 400-mL canisters.

- Vacuubrand Vacuum Pumps, model MZ-2, 120 VAC, 60Hz, 3.0A.

The Entech 4600 dynamic diluter is used to pressurize canisters.

7.2 Reagents

- Helium, ultra-high purity
- Nitrogen, high purity
- Zero Air

8 ANALYTICAL PROCEDURES

Canisters used for soil gas sampling and indoor air sampling are segregated to prevent contamination of the indoor air equipment by the (potentially) high concentration of target analytes in soil gas. Soil gas canisters should not be cleaned in the same batch as indoor air canisters in order to prevent cross-contamination.

Five cleaning cycles are routinely used for all canisters. If all canisters in a batch are known to be "clean" based on the analytical results below the reporting limit, the analyst may use fewer cycles. However, more than 5 cleaning cycles may be necessary depending on the concentration of analytes or contamination present in the canisters. Canisters with analytes whose concentrations have been found to exceed the calibration range of the instrument may require 15-90 cleaning cycles.

8.1 Leak Testing Canisters

See Appendix C regarding leak testing the manifold and canisters prior to connecting canisters to the manifold. Connect 400-mL canisters to the manifold only after system leak testing is complete.

Turn off high vacuum pump by flipping the appropriate toggle switches on the front of the 3100A before loading canisters.

8.2 Cleaning Canisters

8.2.1 Cleaning 6-L Canisters

1. If the canister pressure is above ambient pressure, open the canister valve in a hood to bring contents to ambient pressure then close the canister valve.

   WARNING: Do not connect canisters with pressures above 15 psig (30 psia) to the cleaning system.

2. Turn on the rough pump; wait 1-2 minutes.

3. Connect canisters to the manifold making sure to tighten the connection enough so no leak will occur (finger tight plus an additional 1/2 turn should be sufficient).

4. Leave canister valves closed during leak testing.

5. Open canister valves before starting the cleaning sequence.

6. Turn on high vacuum pump by flipping the appropriate toggle switches on the front of the 3100A. Wait until green light on the high vacuum pump indicates that it is running at approximately 27,000 rpm.

7. Turn on the oven switch and adjust setting (if necessary) so the oven temperature is at 100ºC.

8. Double click on the 3100 icon in the SmartLab Group to start an instrument session. Load the desired method or enter the desired parameters and enter RUN Control Screen. (See Appendix B for method parameters).

9. Press Go in the RUN Control Screen, automated cleaning will occur over the next 1-12 hours depending on the parameters chosen and the number of canisters being cleaned.

   Note: If a final vacuum below 30 mTorr cannot be attained there may be a leak in the canister at a weld or at the valve. Try closing each canister valve one at a time to find the leaking canister. If a canister is determined to be leaking see Appendix C.

10. At the conclusion of the cleaning run, make sure the canister software has been "stopped".

11. Press "All Off" in the RUN Control Screen. Do not remove canisters while the high vacuum pump is on.

12. Turn off oven and allow to cool to ambient temperature.

13. Pressurize canisters to approximately 10 psig (~25 psia) with humidified air or humidified nitrogen by pressing "All Off" in the RUN Control Screen, then alternate between pressing "Diluents" and "All Off" button until a pressure of 25 $\pm$ 2 psia is attained. (Pressure is displayed in the RUN Control Screen under "Pressure (psia).") If over pressurization occurs, press "All Off" in the RUN Control Screen, then alternate between pressing "Rough Pump" and "All Off" button until a pressure of 25 $\pm$ 2 psia is attained. Alternatively, pressurize canisters to approximately 10 psig with humidified zero air using the Entech 4600 dynamic diluter.

14. Close all 6-L canister valves and remove the canisters from the oven.

8.2.2 Cleaning 400-mL or 1 L Canisters

1. If the canister pressure is above ambient pressure, open the canister valve in a hood by attaching a female quick connect to the valve stem and allow to reach ambient pressure.

   WARNING: Do not connect canisters containing pressures above 15 psig (30 psia) to the cleaning system.

2. Turn on the rough pump, wait 1-2 minutes.

3. Connect canisters directly to the manifold using the quick connect fittings.

4. Turn on high vacuum pump by flipping the appropriate toggle switches on the front of the 3100A. Wait until green light on the high vacuum pump indicates that it is running at approximately 27,000 rpm.

5. Turn on the oven switch and adjust setting (if necessary) so the oven temperature is at 100°C.

6. Double click on the 3100 icon in the SmartLab Group to start an instrument session. Load the desired method or enter the desired parameters and enter RUN Control Screen. (See Appendix B for method parameters).

   Note: For all canisters, a minimum of five cleaning cycles is recommended.

7. Press Go in the RUN Control Screen, automated cleaning will occur over the next 1-12 hours depending on the parameters chosen and the number of canisters being cleaned.

Note: If a final vacuum below 30 mTorr cannot be attained, there may be a leak in the canister at a weld or at the valve. Try removing each canister one at a time to find the leaking canister. If a canister is determined to be leaking, refer to Appendix C.

8. At the conclusion of the cleaning run, make sure the canister software has been "stopped".

9. Press "All Off" in the RUN Control Screen.

10. Turn off oven and allow cooling to ambient temperature.

11. Pressurize canisters to approximately 10 psig (~25 psia) with humidified zero grade air or humidified nitrogen by pressing "All Off" in the RUN Control Screen, then alternate between pressing "Diluents" and "All Off" button until a pressure of 25 $\pm$ 2 psia is attained. (Pressure is displayed in the RUN Control Screen under "Pressure (psia).") If over pressurization occurs, press "All Off" in the RUN Control Screen, then alternate between pressing "Rough Pump" and "All Off" button until a pressure of 25 $\pm$ 2 psia is attained.

12. Remove the canisters from the oven.

8.3 Cleaning Restrictors and Flow Controller Inlet Parts

Place restrictors and/or flow controller inlet parts (filter, filter tube, and restrictor), in a 1 L sampling jar equipped with a quick connect fitting.

Connect jar directly to the manifold using the quick connect fittings, heat to 100°C, and clean for a minimum of five cycles.

Alternatively, place the restrictor and or flow controller inlet parts on the floor of the oven, heat to 100°C, and clean for a minimum of 5 minutes.

8.4 Canister Certification

Canisters are routinely analyzed and certified individually but may be certified per cleaning batch. The method and target analytes of interest can be determined on a project-specific basis or per client's request. When performing "batch certification", a canister is randomly selected from the cleaning batch and analyzed.

Following EPA Region 9 Laboratory SOP 311 or 314, analyze an aliquot of gas from each canister or batch. If any target compounds are detected at a concentration greater than or equal to half the quantitation limit, re-clean and re-analyze the canister or batch.

Note: Dichloromethane and acetone are common laboratory contaminants; re-cleaning and re-analysis is necessary if the contaminant is required for the project. Note contamination in the Canister Database in G:\USER\ESAT\1 Organic Group\AIR\R9CanisterDB R4.mdb. See Appendix D.

If the cleanliness checks are satisfactory, record the date that the canisters were cleaned and the date the canisters were certified in the Canister Database. See Appendix D.

Note: Canister certification is valid for 6 months; canisters on the shelf for longer than this period must be re-certified. If, upon re-certification, the cleanliness check is not satisfactory the canister must be re-cleaned, leak tested, and re-certified.

Evacuate the canisters to <30mTorr and mark the gauge. Store canisters in this evacuated condition until they are ready to use. Store the canister on the storage racks in Room 203. Check the pressure after a few days. If the pressure has changed, fix the leak. Leaking canisters must be re-cleaned and re-certified.

8.5 Flow Controller Calibration

Refer to Appendix C for details on flow controller flow adjustments and cleaning.

8.6 Pre-shipment Procedures

Canisters must not leak and flow controllers must be working properly before they are shipped.

To detect any leaks in the can, 6-L cans are evacuated after certification and stored under vacuum for a minimum of one day prior to shipping. Verify that the pressure indicated on the gauge has not changed.

To verify proper operation of flow controller:

1. Attach the controller to an evacuated can.
2. Plug the flow controller inlet.
3. Open the can valve momentarily to evacuate the flow controller body.
4. Close the can valve to isolate the flow controller.
5. Monitor the flow controller gauge for a period of five minutes to check for a leak.
6. If the gauge moves less than 5 in. Hg, the controller is good.
7. If the gauge moves more than 5 in. Hg, the controller is bad (i.e. the intrusion of air into the can from locations other than the inlet will significantly impact total volume collected).

8.7 Maintenance

Fill the zero air humidifier reservoir no more than half full with organic free water that has been prepared according to EPA Region 9 Laboratory SOP 205 and attach it to the back of the 3100A controller with the water level indicator facing away from the instrument for easy viewing. Filling the humidifier more than half full may cause liquid water to be transferred into the cleaning manifold, making it impossible to reach high vacuum.

9 QUALITY CONTROL

Canisters are certified as clean provided they meet the QC criteria specified in EPA Region 9 Laboratory SOP 311 and/or SOP 314.

10 DOCUMENTATION

10.1 Canister Certification Data

The requirements for a complete data package for the determination of volatile organic compounds in air are outlined in EPA Region 9 Laboratory SOP 311 and SOP 314. As indicated in those SOPs, the data package must include documentation on the cleaning and certification of canisters used to collect air samples or to prepare dilutions of air samples. Include relevant pages from the Canister Database printouts, the raw data quantitation report, the reconstructed ion chromatogram (RIC) of the data file, and enhanced spectra of non-target compounds (for TO15 full scan only) detected in the canister with a library search listing the three best fits of a forward library search of the non-target compounds.

10.2 SOP Distribution and Acknowledgement

After approval, distribute an electronic copy of the final SOP to all laboratory staff expected to perform the SOP or review data generated by the SOP. (The Lab QC Database contains a list of assigned analysts for each SOP). All approved EPA Region 9 Laboratory SOPs are maintained on the local area network or on the Lab QA SharePoint site in Adobe Acrobat portable document format.

Analyst training is documented via the Training Record form and the Read and Understood Signature log; the latter is entered into the Lab QC Database.

10.3 SOP Revisions

Revisions to this SOP are summarized in Appendix E.

11 REFERENCES

EPA Region 9 Laboratory documents (SOPs, the Laboratory Quality Assurance Plan, etc.) are not included in this list. Analysts are referred to the SOP database on the local area network, LIMS, or the Lab QA SharePoint site for these documents; laboratory users should contact the Chemistry Team Leader or Laboratory QAO for copies of any supporting documents.

Entech, 3100A canister cleaning system operator′s manual.

USEPA Compendium Method TO-14A; *Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using Specially Prepared Canisters with Subsequent Analysis by Gas Chromatography. January 1999.*

USEPA Compendium Method TO-15; *Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS); January 1999.*

USEPA Compendium of ERT Air Sampling Procedures. May 1992.

USEPA *Statement of Work (SOW) for the Analysis of Air Toxics from Superfund Sites. Draft report, June 1990.*

APPENDIX A.
DEVIATIONS FROM THE REFERENCE METHOD

1. Method TO-15 defines cleanliness as less than 0.2 ppbv of targeted VOCs. This SOP defines cleanliness as less than reporting limit (1/2 the quantitation limit) of targeted VOCs (see respective SOPs for quantitation limits).

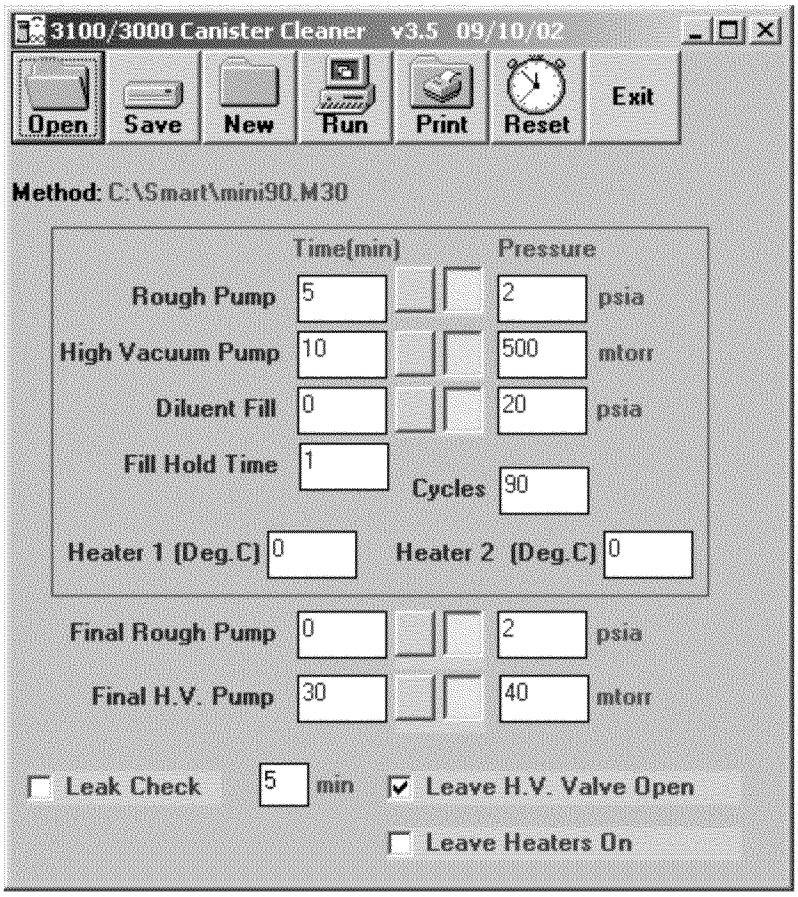
2. Method TO-15 specifies that canisters be pressurized to 30 psig prior to analysis and certification. This SOP specifies that canisters be pressurized to 10 psig (25 psia) prior to analysis and certification. The positive pressure at 10 psig (25 psia) prevents contamination in case of a leak and gives a more accurate measurement of the canisters' cleanliness. Most samples received at this laboratory are ambient samples with pressures at or below 0 psig and are pressurized to approximately 10 psig (25 psia) prior to analysis.

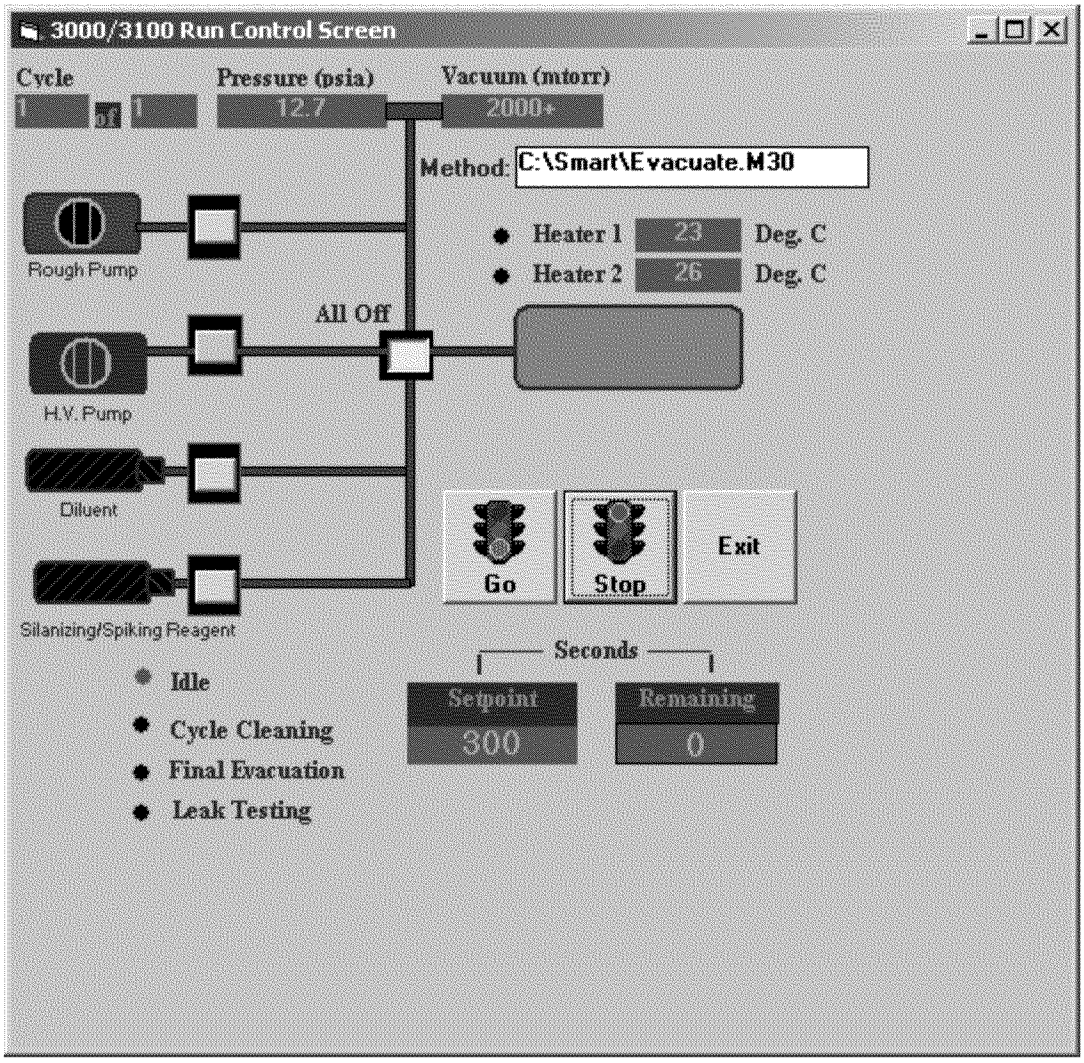
APPENDIX B.
METHOD PARAMETERS

The following parameters may be changed to optimize run time and attain the desired level of cleanliness:

1. Rough Pump (Time): Length of rough pump isolation valve engagement cycle, normally 5 minutes.
2. Rough Pump (Pressure): Pressure to be attained at the end of the rough pump cycle, normally 2 psia. The inability to reach this pressure indicates a leak.
3. High Vacuum Pump (Time): Length of time of the high vacuum pump engagement cycle.
4. High Vacuum Pump (Pressure): Pressure to be attained at the end of the high vacuum pump cycle, normally 500 mTorr.
5. Diluent Fill (Time): Maximum time needed to reach specific pressure at the end of the diluent fill cycle. The need for longer time period indicates a leak.
6. Diluent Fill Pressure: Pressure to attain at the end of the diluent fill cycle.
7. Fill Hold Time: Time to hold pressure at the end of diluent fill cycle. **This parameter is normally set for 1-2 minutes to allow contaminants residing on inside walls of the canister to reach equilibrium with the gas (diluent) phase. This is an important parameter to achieve maximum cleanliness of the canister.**
8. Cycles: Number of cleaning cycles to perform.
9. Heater: Temperature of oven (this is a monitor not a controller). Temperature is set using a dial in front of the oven. Normally 100°C.
10. Final Rough Pump (Time): Length of rough pump isolation valve engagement at the end of the cycle normally set to 0 minutes since the pressure is maintained by the high vacuum pump.
11. Rough Pump (Pressure): Pressure to be maintained at the end of the cleaning cycle, normally 2 psia.
12. Final High Vacuum Pump (Time): Time to maintain specific pressure for at the end of the cycle.
13. Final High Vacuum Pump (Pressure): Pressure to be maintained at the end of cycle, normally 10-50 mTorr.
14. Leak Check: Not used
15. Leave H.V. Valve Open: Checked on. This allows the H.V. isolation valve to remain open so that the final pressure of 10-50 mTorr is maintained otherwise, nitrogen or ambient air may re-enter the canister.
16. Leave Heaters on: Not used.

Typical Method:





APPENDIX C.
LEAK TESTING OF CANISTERS AND FLOW CONTROLLERS

Leak Testing Canister Cleaning System

The 3100A canister cleaner should be leak tested after installation and periodically to insure leak-free operation. Using the built-in pirani gauge, leaks can be spotted quickly by monitoring the rise in system pressure after evacuation. Leaks can also be detected by selecting the manual FILL button and then monitoring the pressure on the 0-50 psia readout while the system is in standby. The smaller the volume in the manifold, the greater the pressure change, so small leaks are more easily detected when the canister valves are closed or when canisters are removed altogether.

1. For the 6-L canister cleaning system, plug the openings on the manifold with 1/4" caps to generate a closed system. This step is not necessary for the 400-mL canister system since the individual canister ports are normally closed.
2. Turn on the 3100A controller and click on Rough Pump button.
3. Wait until the Vacuum (mTorr) pressure falls to 2000 mTorr (2 psia).
4. Click on H.V. Pump button. The system high vacuum reading "Vacuum (mTorr)" should very quickly drop below 2000 mTorr. The inability to reach below 50 mTorr within 5 minutes indicates either a system leak or the presence of liquid water in the lines.

Note that if liquid water exists, the lines will get very cold where the water is evaporating due to its large heat of vaporization (540 cal/g). A second clue that water may be in the lines is if the system holds pressure (35-40 psia) with no change in 10 minutes. If water is in the lines, make sure the humidifier level is less than 50%, then perform an automated 50 cycle fill and evacuation, just spending 1 minute on pump 2 (high vacuum) during each cycle.
An actual leak exists if the system cannot reach high vacuum and cannot hold pressure. The location of a leak can best be found by pressurizing the system with helium and using an electronic leak detector. If vacuum leak checking was done while canisters were attached, be aware that the symptoms of a leak can actually be the result of a leak through the canister valve. While the system is evacuating using the high vacuum pump (pump 2), try gently tightening each canister Nupro valve and see if there is any change in the high vacuum reading. Nupro valves which require too much force to close completely may have to be replaced to insure leak-free field sampling. After finding and stopping the leak, repeat the vacuum hold test to confirm an air-tight system.

Pressure Testing Canisters (6-L, 1-L, and 400-mL)

Summa canisters should be leak tested periodically to insure leak-free operation.

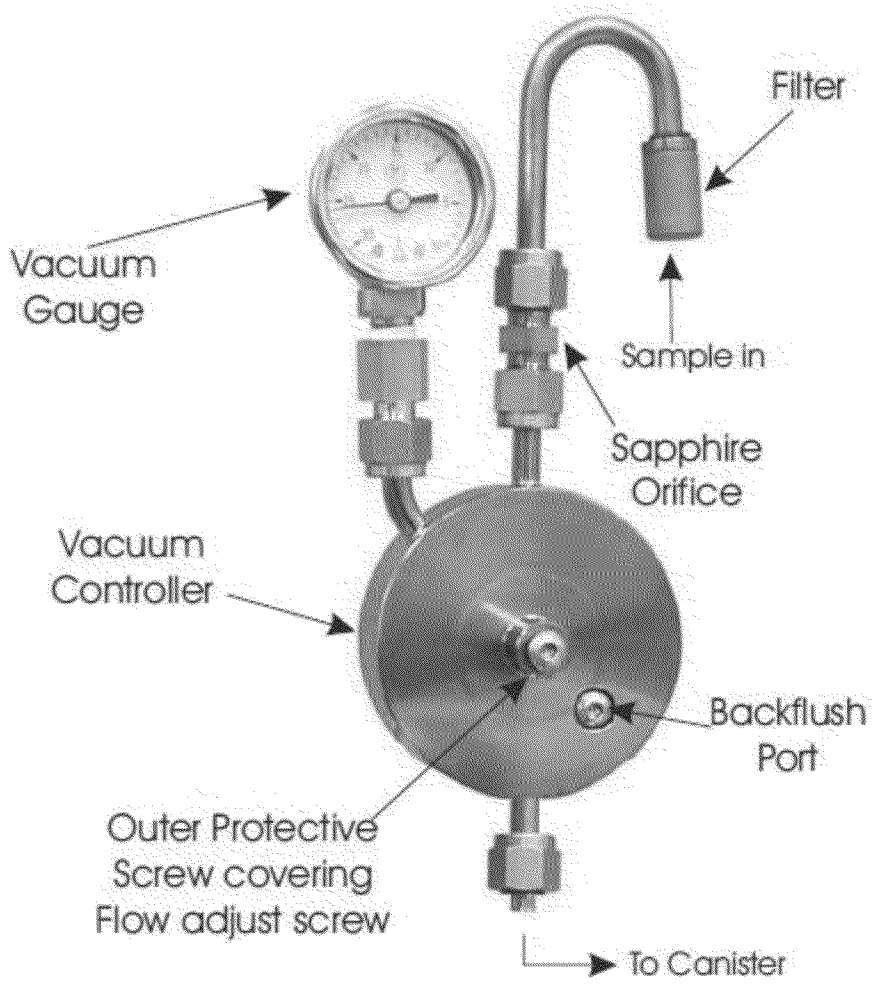
1. Pressurize the canister to 30 psig and record the initial time, date, and exact pressure.
2. For 1-L or 400-mL canisters, immerse the entire stem and valve in tap water to identify gross leaks, let stem soak for 2-3 minutes recheck the canister for leaks then wipe out excess water.
3. Allow canisters to stand overnight (24 hours) and record the final time, date, and pressure.
4. A pressure difference greater than 2 psig verifies the presence of a leak.
5. If a canister fails the pressure test, update the Canister Database and indicate that the canister in question is "possible leaker/damaged."
6. Place a tag on the 6-L canister indicating that it has a leak.
7. One common location for leak is the ¼" O-ring below the valve or the quick-connect valve itself.
8. If possible, determine the cause of the leak and repair it.
9. Retest all repaired canisters.
10. If the canister passes the pressure test, prepare it for immediate vacuum testing, cleaning, and re-certification or update the Canister Database to "To be cleaned."

Leak Testing 1-L or 400-mL Canisters

Summa canisters should be leak tested periodically to insure leak-free operation. Using the cleaning system built-in pirani gauge, leaky canisters can be spotted quickly by monitoring the rise in system pressure after the evacuated canister is attached to the system and monitoring the pressure on the 0-50 psia readout while the system is in standby.

1. Test the cleaning system for leaks using the procedure outlined above. Proceed to step 2 when the system passes the test.
2. Perform the test using only canisters that have passed the pressure check.
3. Attach the canisters to be tested in the cleaning oven.
4. Turn on the 3100A controller, in the "3000/3100 Run Control Screen," and click on Rough Pump button.
5. Wait until the Vacuum (mTorr) pressure falls to 2000 mTorr (2 psia).
6. Click on H.V. Pump button. The system high vacuum reading "Vacuum (mTorr)" should very quickly drop below 2000 mTorr. The inability to reach below 50 mTorr within 5 minutes indicates the presences of leaky canister(s).
7. Evacuate the canisters to be tested to less than 50 mTorr.
8. Remove the canisters from the system while the system is under vacuum (i.e. H.V. Pump is clicked on).
9. Let the canisters stand for about 1 hour.
10. Verify that system high vacuum reading "Vacuum (mTorr)" is below 50 mTorr.
11. Attach the first canister to be tested to the oven.
12. The vacuum reading should not rise by more than 10 mTorr and should fall back quickly to the original reading. Higher reading and or failure to drop back indicates a leaky canister.
13. Remove the canister and repeat steps 10 and 11 for the remaining canisters.
14. Update the Canister Database.

FLOW CONTROLLER (CS1200) FLOW ADJUSTMENTS AND CLEANING



Principle of Operation

The CS1200 is a high purity flow regulation system used to fill canisters at a constant rate from vacuum to within 1 psi of atmospheric pressure without requiring power. The CS1200 consists of 2 main parts; the vacuum controller body and the restrictor. The vacuum controller maintains a -0.3 to -1 psi pressure differential relative to atmospheric pressure no matter what the vacuum is on the outlet. By changing the value of the restrictor on the inlet, different flow rates, or canister fill rates can be achieved. For any given restrictor, the flow rate can only be changed by a factor of 2-3x. This is done by adjusting the 1/8″ hex set screw on the vacuum controller body. The following table gives the appropriate restrictor for different applications (target pressure is 0.9 atmospheres).

Restrictor PN	Stamp Code	400-mL Canister (fill time in minutes)	1 L Canister (fill in minutes)	6-L Canister (fill in Hours)	Target Flow Rate (ccm)*
39-23010	1	2	8	1	80
39-23030	2	10	25	3	27
39-23080	3	30	75	8	10
39-23080	3			12	6.7
39-23080	3			16	5.1
39-23240	4	2 hr	5 hr	24	3.4
39-14010	5	8 hr	16-24 hr	1 week	0.5

* - Multiply this value by Pa/Po where Pa is local atmospheric pressure and Po is standard Pressure (760 torr). This will prevent premature filling of the canister during higher elevation sampling (example – Denver, CoO)

Calibrating Flow with Flow Meters

Calibration requires attachment of the Entech Flow Calibrator (PN 39-20020 10 ccm, or 39-20035 100ccm, or equivalent) to the inlet while the outlet is connected to a canister under vacuum.

Follow the procedure below:

1. Connect the correct restrictor to the inlet side of the CS1200 flow controller as per Table above.
2. Connect the outlet to a 6-L canister that is at a vacuum of -15 to -30 psi.
3. Connect a flow meter to the inlet (below the filter).
4. Open the canister valve to start flow and close again. Check if pressure is holding. If the pressure drops, retighten all nuts and check again. Hold for five minutes to make sure pressure is holding.
5. If there is no leak, open the canister valve to start flow.
6. Adjust the set screw found under the tamper proof nut so that the flows agree (to $\pm$ 5%) with table above.

Note that very little adjustment should be necessary and flows should never be more that 2-3 turns lower or higher than the desired set point if the correct restrictor is installed. IF 2-3 TURNS DOES NOT RESULT IN A FLOW OR FLOW CHANGE, STOP AND CHECK THE FLOW CALIBRATOR FOR PROPER OPERATION. THE INTERNAL DIAPHRAGM MAY BE DAMAGED BY OVERTIGHTENING!

Cleaning the CS1200

The filter and filter tube orifice (restrictor) should be cleaned routinely by following Section 8.3, Cleaning Restrictors and Flow Controller Inlet Parts, of this SOP.

Cleaning the CS1200 Vacuum Controller

The CS1200 Vacuum Controller can be cleaned quickly by removing the screw covering the

back flush port and connecting nitrogen or zero air at 2-3 psig at the outlet for back flushing. For ambient applications, 1-2 minutes of flushing will be sufficient.

If the CS1200 was used to sample VOCs at high concentrations (10 ppm or higher), it may be necessary to mount the CS1200 inside an oven while heating to 70°C. Several CS1200s can be cleaned simultaneously by constructing a manifold using 1/4“ stainless steel fittings.

Troubleshooting Damaged Diaphragm

Damaged may be caused by over-tightening of the adjustment screw and can lead to a lack of control of flow rate or inability to adjust the rate.

Tools Needed: 1/8 Hex Key, 5/64” Hex Key, Forceps, Retaining Ring Pliers and Safety Glasses.

1. Remove adjustment port cover screw and back flush port screws. Turn the adjustment screw counter clockwise until it stops.
2. Place the flow controller body on table with the adjustment screw on bottom. Loosen set screws (x4) on back of the CS1200. Insert the retaining ring pliers into the retaining ring holes and squeeze the pliers to compress the ring. When adequately compressed, the ring should pop out of the controller body.
3. Flip the flow controller body over, so the adjustment screw is facing up to remove the cover plate and metal disc.
4. Examine the diaphragm. A damaged diaphragm surface will appear deformed, creased, or wrinkled. If it does not appear damaged proceed to step 9.
5. Gently pry the edges of the diaphragm up with forceps and invert the controller body to remove the diaphragm.
6. Inspect to make certain that the large O-ring is seated snugly in the groove inside the flow controller body. Replace if cracked or dry.
7. Ensure that the tiny O-rings are in the center of the adjustment screw. Replace if cracked or dry.
8. Clean the inner body with methanol if necessary. Wipe dry.
9. Insert a new diaphragm into the flow controller. The raised edge will be seated on the O-ring.
10. Place the metal disc on top of the diaphragm with the flat side facing up (concave side down) and place the back plate on top with the flat side facing down (raised surface with screw holes up).
11. Install the retaining ring by compressing it with the pliers. Make sure that the ring expands into the inside groove and snaps tight. Finally, evenly tighten all the 4 set screw to compress the lip of the diaphragm to the O-ring.
12. Recalibrate the flow controller. Remember to avoid turning the adjustment screw more than 5 turns clockwise .

Filter Replacement and Restrictor Replacement

Replace filter and restrictor if contaminated or if buildup of particulates at restrictor orifice is noted.

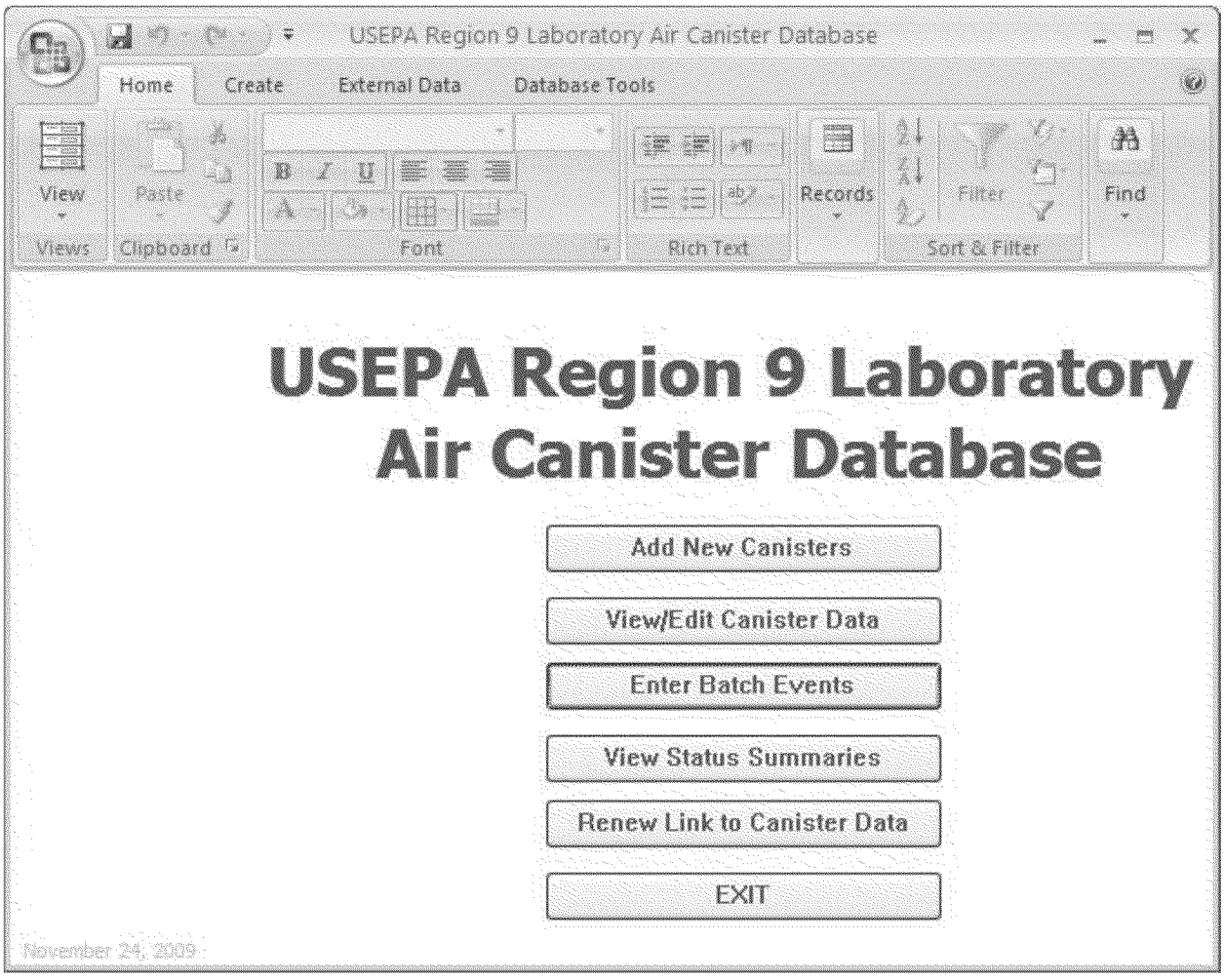
APPENDIX D.
CANISTER DATABASE

The following parameters are used throughout the database:

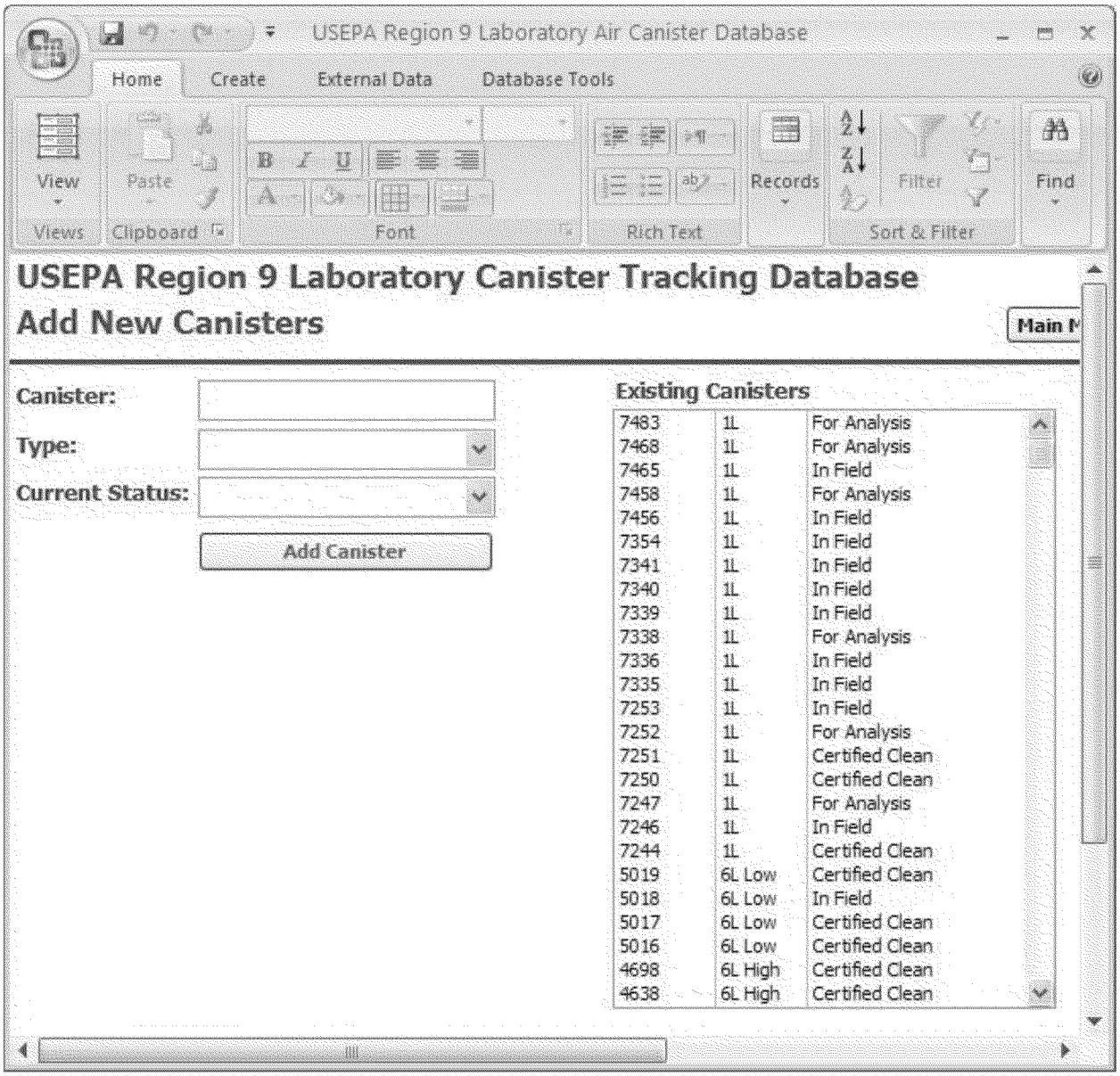
Canister:
- Canister Serial Number
- Flow Controller Serial Number

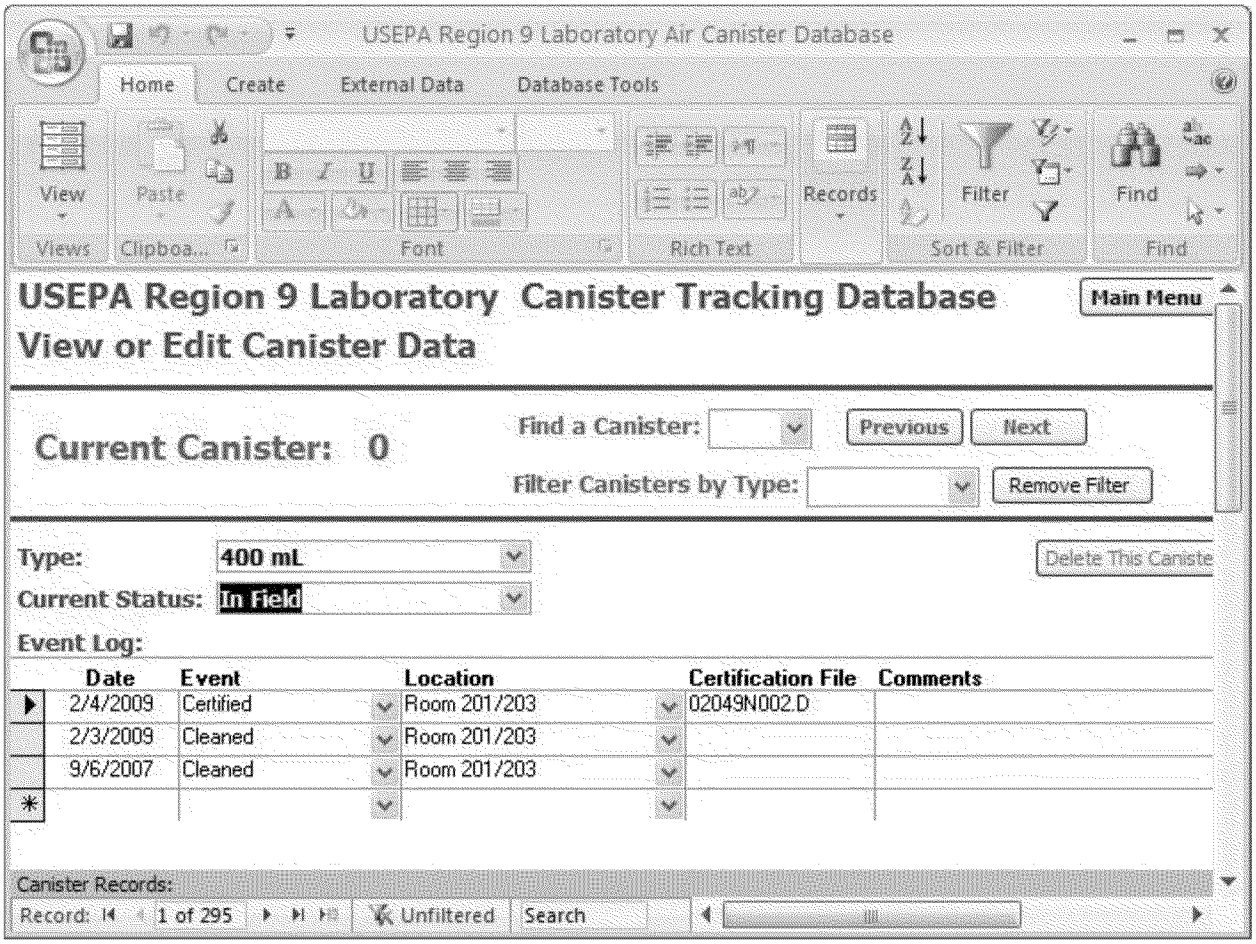
Type:
- 400-mL
- 6-L High (Full Scan Canisters)
- 6-L Low (SIM, Ambient air, Canisters)
- 1-L
- Flow Controller

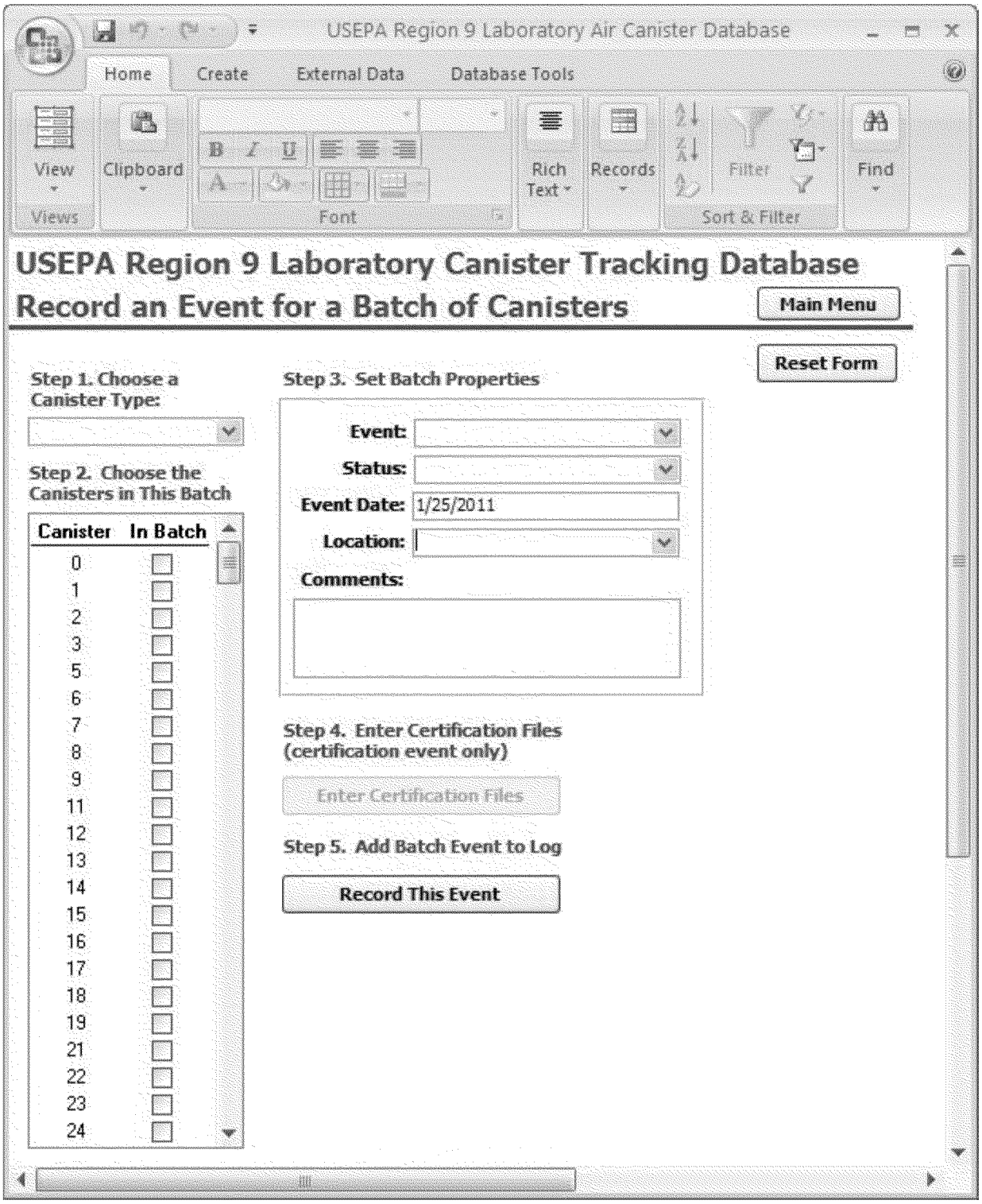
Current Status:
- Clean Ready to Certify
- Certified Clean
- Batch Certified Clean
- In Field
- For Analysis
- To Be Cleaned
- Damaged
- Lab Use



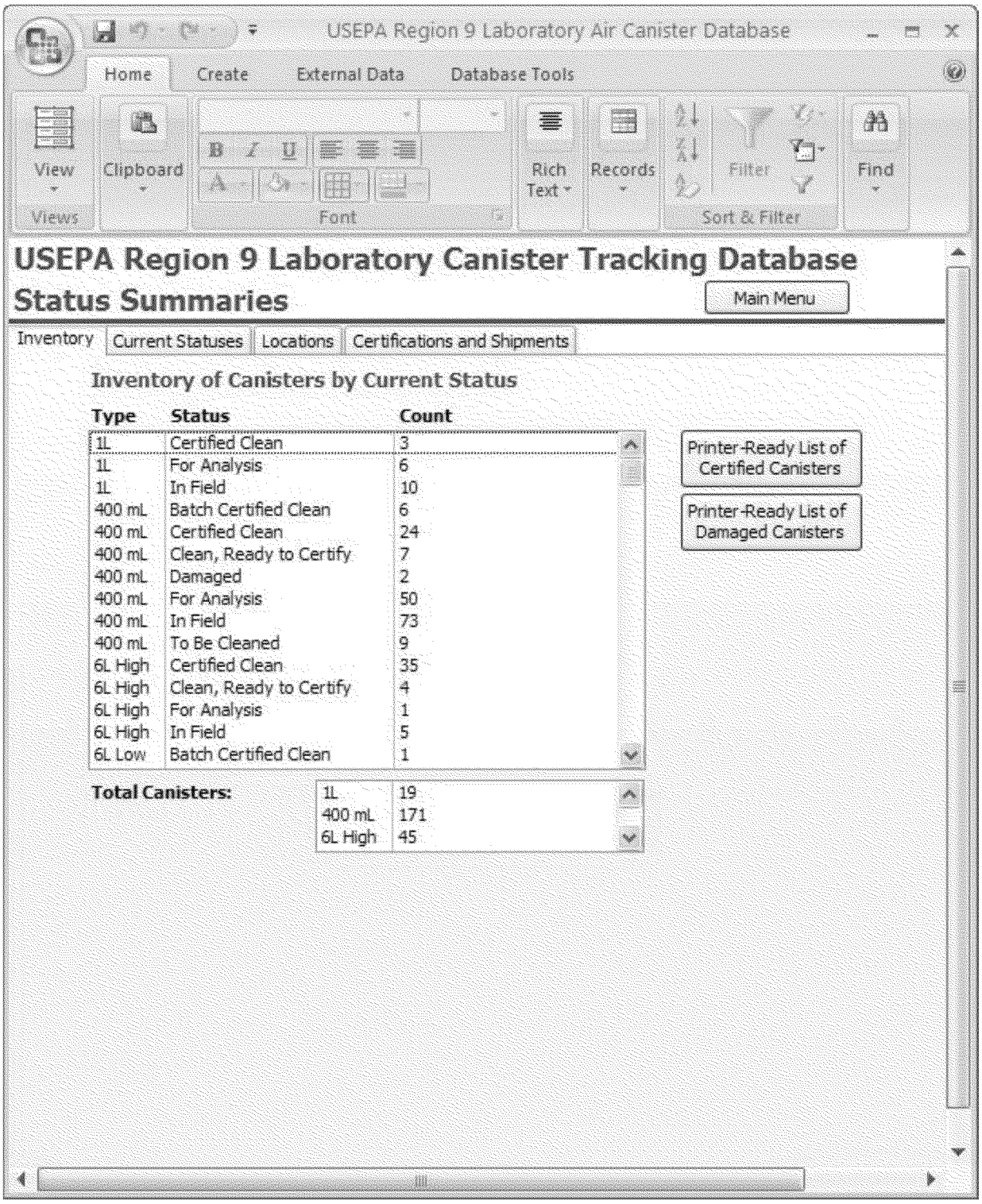
Add New Canister

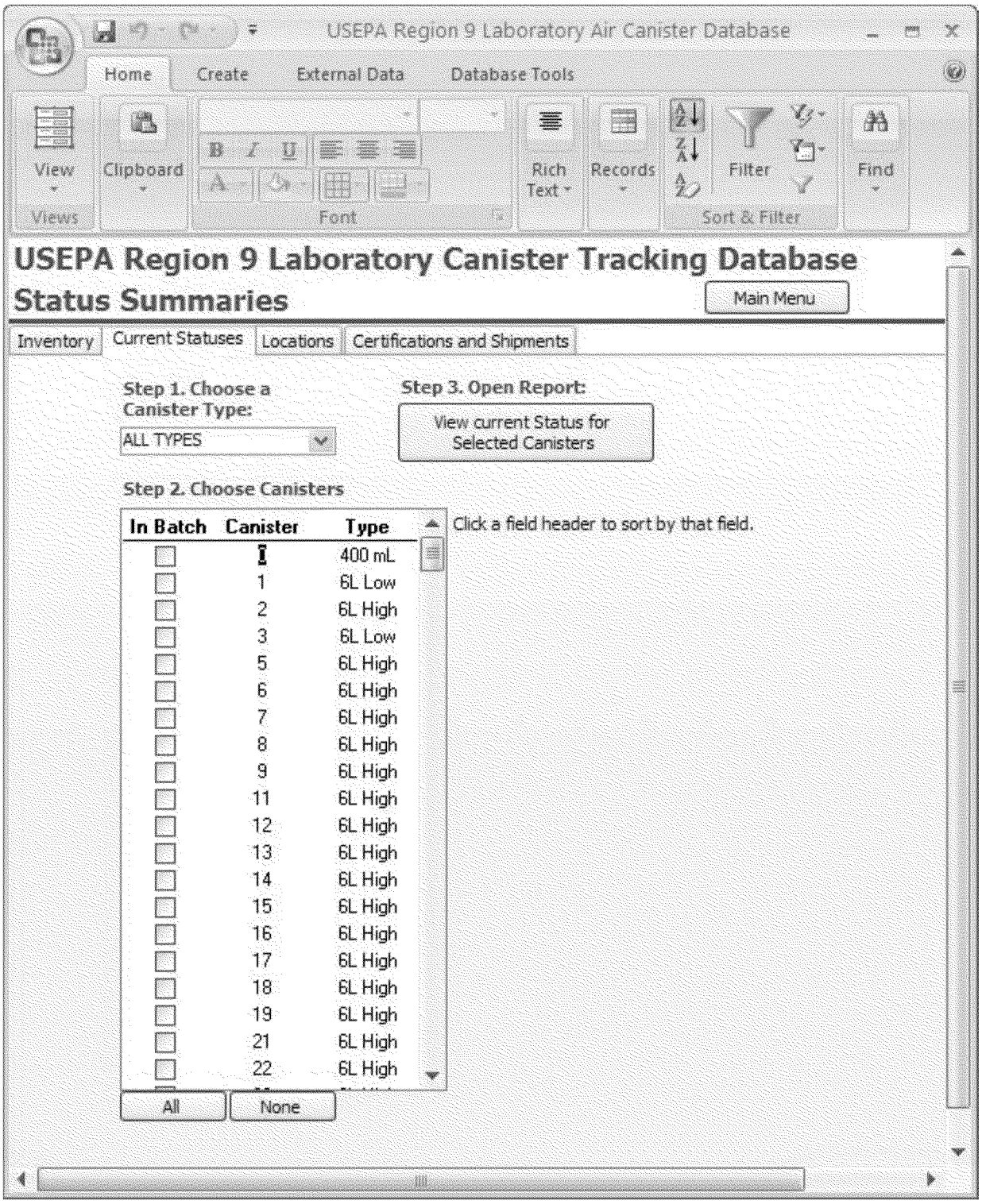


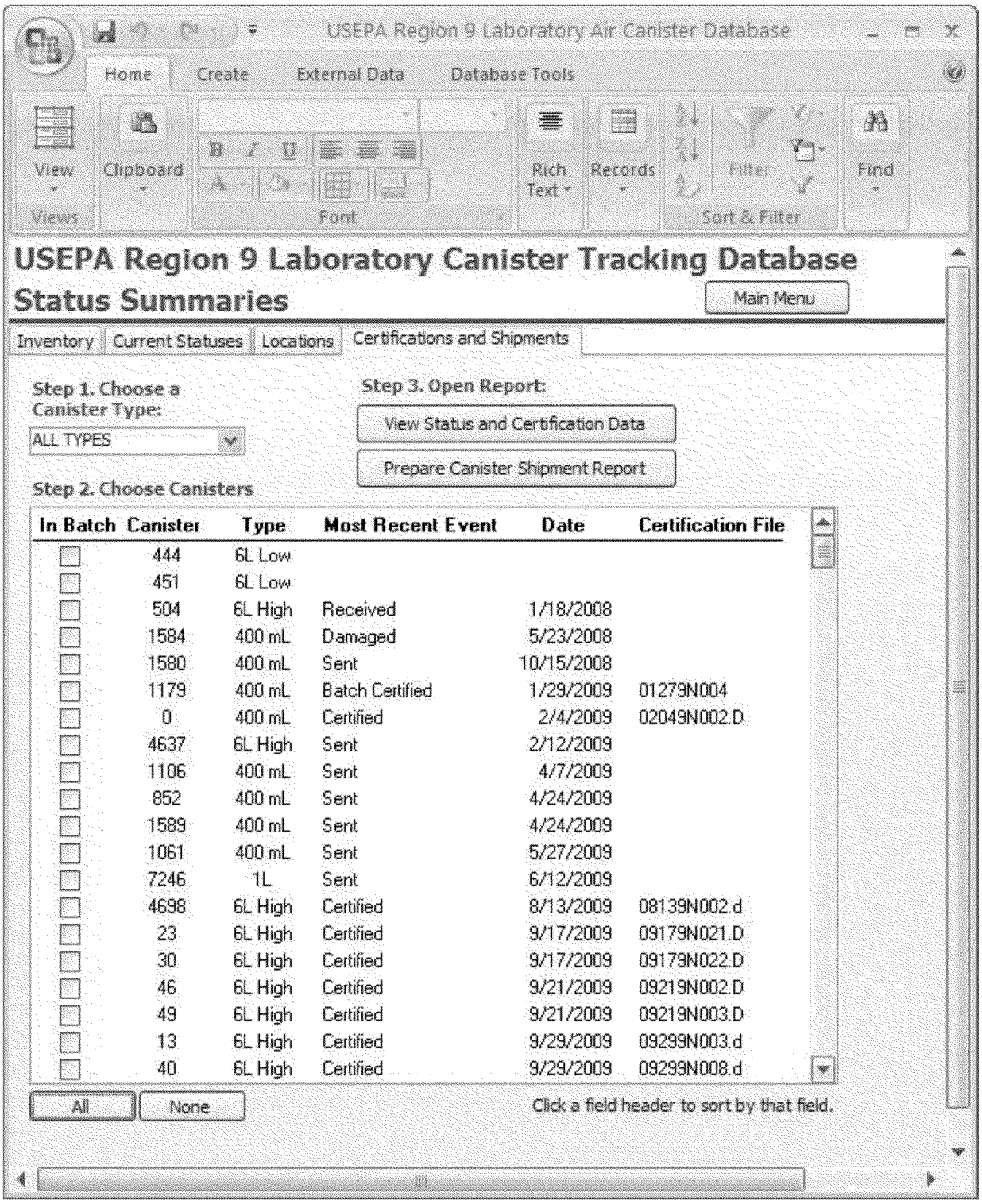




Click Canister serial number(s) and appropriate event





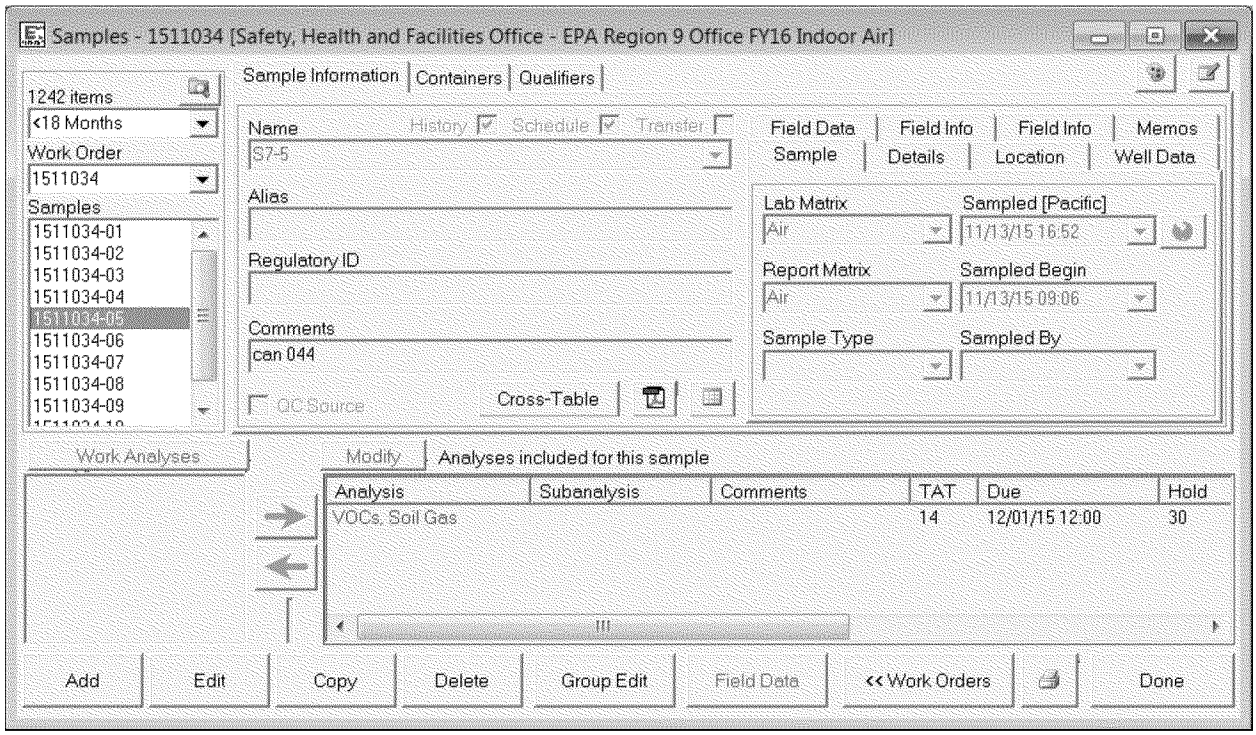


Recommended Canister Certification Documentation Procedure:

The laboratory is implementing a plan to store canister certifications in the Element LIMS. Although the specifics may change, the general concept is as follows.

Canister certification files, which are identified in the Canister Database on the shared network drive, are copied to a shared location. A PDF of the analytical results is created and stored on the LAN at I:\Can Certs\ by canister number. The filename of the PDF must be the canister number. When canisters are re-certified, the existing files will be overwritten.

The canister certification is attached in LIMS at sample login or later by clicking on the PDF icon in the middle of the Sample screen (see below) and uploading the PDF file.



This process associates one canister certification with each use of the canister to collect a sample.

APPENDIX E.
REVISION HISTORY

STANDARD OPERATING PROCEDURE: 312
Revision: 5, Effective: 01/15/16

MAINTENANCE AND TRACKING OF CANISTERS AND FLOW CONTROLLERS

Revision	Effective Date	Description
4	4/22/11	1. Added cleaning and certification of flow controllers. 2. Added Canister Tracking Database to replace manual logs.
5	01/15/16	1. Revised SOP title to reflect all activities. 2. Added procedures to check canisters and flow controllers for leaks. 3. Added recommended canister certification documentation procedure to Appendix D.